

# Ampoule Failure Sensor Time Response Testing — Experiments 2 and 3

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## **NASA TECHNICAL MEMORANDUM**

### **AMPOULE FAILURE SENSOR TIME RESPONSE TESTING EXPERIMENT 2 AND EXPERIMENT 3**

#### **INTRODUCTION**

Numerous ampoule failure sensor tests were successfully completed prior to this series of time response experiments [1,2]. These experiments proved the ampoule failure sensor concept and eventual design along with their durability as they were subjected to semiconductor materials at temperatures up to 1260 °C. Experiment 2 and Experiment 3 is a continuation of the ampoule failure sensor time response testing conducted in Experiment 1 [3]. These experiments were configured to measure the response time of the ampoule failure sensor upon a known breach of an ampoule containing gallium-arsenide (GaAs) at its processing temperature. This technical report will discuss the experiment objectives, pre-experiment obstacles, experiment configuration, results, and conclusions.

#### **EXPERIMENT OBJECTIVES**

Based on the results of Experiment 1, the wire diameter of ampoule failure aensor was changed from 0.003 inch to 0.005 inch to increase the durability of the sensor. The primary objective of Experiment 2 was to measure the response time of the larger ampoule failure sensors when exposed to GaAs at a temperature of 1260 °C. The secondary objective of this experiment was to measure the time it would take the GaAs to breach the WC-103 silicide coated containing cartridge.

The objectives of Experiment 3 were identical to those of Experiment 2, but contained sensors that were built to flight specifications. This new flight configuration will be discussed later.

#### **AMPOULE FAILURE SENSOR AND EXPERIMENTAL CONFIGURATION**

The sensor developed takes advantage of the high-temperature chemical reaction between the semiconductor material and the sensor material. The ampoule failure sensor configurations for Experiment 2 and Experiment 3 are shown in Figure 1a and 1b, respectively. The elements are made of 0.005 inch diameter pure platinum wire. Upon ampoule failure, the sensor is immediately exposed to the molten semiconductor material and the chemical reaction causes a resistance change on the order of megohms. Therefore, the resistance is monitored to detect an ampoule failure.

These experiments, as well as previous experiments, were conducted at the Marshall Space Flight Center in the Hazardous Operations Facility, building 4475. The experiment configuration is discussed in detail in ref. [2]. Two processing furnaces were used in these experiments. The primary furnace was a 16 inch platinum-40% rhodium element furnace used to heat the sample up to the GaAs processing temperature of 1260 °C and the secondary furnace was a 12 inch nichrome wound furnace with 9 inches of the 21 inch alumina core tube unwound. This furnace was used to pre-heat the sample to 1000 °C. The secondary furnace sat upon and extended 9 inches into the primary heater core. Figure 2 shows the experimental arrangement in the fume hood.

The ampoule failure sensors used in these experiments are shown in Figure 1. They consist of a two-hole alumina protection tube (0.044 inch) with a machined flat area in which only one hole remains. The sensor wires were extended out from the holes and beaded or a single element was threaded through one hole and back into the other hole. The machined area provides a larger gap between the two wires for the chemical reaction. The sensor design used for Experiment 2 is shown in Figure 1a. This design maximized the free surface area of the platinum wire. The single loop conductor configuration, shown in Figure 1b, is the flight configuration and was used in Experiment 3. This design was chosen for flight due to space constraints within the flight cartridge where the helical wrapped design would be in contact with the cartridge wall resulting in possible false readings. Where space and contact area is not critical, the helical wrapped configuration is optimum. For III-V compounds, a pure platinum wire was chosen based on the reaction of platinum and arsenic at elevated temperatures which forms a low melting eutectic. The failure sensors used wire diameters of 0.005 inch. This increase in wire diameter increases the failure sensor's endurance to high temperatures with only a minimal increase in reaction time.

Figure 3 shows the ampoule design that was derived through experimentation. In order to know the exact time of ampoule failure, an ampoule was designed with a thin, angled fused silica tip, which included a flaw. This fused silica tip was attached at the base of the ampoule. When the ampoule is dropped, the tip breaks, allowing the molten semiconductor material to escape from the ampoule.

The ampoules were placed in flight WC-103, silicide coated, cartridges of the Crystal Growth Furnace (CGF). The ampoules were suspended in the cartridges by a nichrome wire until the processing temperatures were achieved at which time the ampoules were released. Two failure sensors in Experiment 2 and three flight configuration sensors in Experiment 3 were potted in an end cap and also placed in the cartridges. The sensors in Experiment 2 were positioned 11 inches from the end cap assembly. In Experiment 3, the sensors were mounted in 1 inch increments, starting at a location of 15 inches from the end cap. Two failure sensors of the helical wrapped design were placed on the exterior of the cartridges to determine when the cartridges breached. Four additional thermocouples were mounted outside the cartridges and utilized for furnace control and monitoring. Figure 4 shows the pre-drop configuration for Experiment 3. Experiment 2 used a similar configuration with fewer internal mounted failure sensors.

The cartridges were placed into the furnaces, pumped down, and then back-filled with argon. The primary furnace was heated to 1260 °C and the secondary furnace was heated to 1000 °C. Upon achievement of the set temperatures, the argon flow was turned off and the ampoule support wire was cut, releasing the ampoule. In Experiment 2 the feed through hole for the ampoule drop wire was left open and in Experiment 3 it was sealed once the ampoule was dropped.

## RESULTS

The results of Experiment 2 and Experiment 3 showed excellent agreement. In Experiment 2, the failure sensor utilized was a double element, platinum wire, helical wrapped and beaded along a machined flat. The ampoule was dropped at 290 minutes into the primary furnace at 1260 °C. Figures 5 and 6 show the time response of the failure sensor. The dropping of the ampoule caused a slight temperature drop on the primary furnace which was expected due to the thermal mass of the ampoule. This temperature drop is a positive indication that the ampoule has successfully dropped into the lower furnace. Three and a half minutes later the sole ampoule failure sensor, INTFS, detected the breach. The experiment continued processing at 1260 °C for 185 minutes after the ampoule drop. During this time the GaAs was reacting with the WC-103 cartridge material. The two failure sensors mounted on the outside of the cartridge did not indicate a failure at any time during this experiment. For this reason, they are omitted from the figures for clarity. After the 185 minute processing time, the lower furnace was shut off in order to cool the cartridge.

After removing the cartridge from the furnace, a visual inspection revealed two small breach points and areas of bubbling under the silicide coating. A minimal amount of GaAs was present on the exterior of the cartridge. This amount was too small for reaction with the outer mounted failure sensors. Most of the arsenic was able to escape through the feed-through hole used for dropping the ampoule which reduced the amount available for reaction with the outer failure sensors. This hole was closed after ampoule dropping in Experiment 3.

In Experiment 3, the three internal mounted ampoule failure sensors were single element, platinum wire type. One of these three failure sensors, BOTFS3, failed prematurely. After the ampoule was dropped, the entire cartridge shifted downward into the furnace by 2 inches. This allowed the failure sensor transition to overheat and degrade the transition between the failure sensor and lead-out wires causing the premature failure. In future tests, the small diameter wires will be brought out to the connector, eliminating this problem. The other two failure sensors operated as expected. The ampoule was dropped at 225 minutes. Figures 7 and 8 show the temperature fluctuations of the primary furnace profile indicating the successful drop and the response time of the failure sensors. Five and a half minutes later both failure sensors indicated the ampoule breach. The experiment was allowed to continue for 125 minutes after the ampoule drop. At this point, the

cartridge was removed from the furnaces and allowed to quickly cool. The failure sensors mounted on the outside of the cartridge did not indicate a cartridge breach and are omitted from the figures for clarity.

Once cooled, the cartridge was inspected for possible breach points. A visual inspection did not indicate any breach points. The Crystal Growth Furnace developer, Teledyne Brown Engineering, also inspected the cartridge. Aided by the removal of some of the silicide coating, they were able to discover a breach in the cartridge. Figure 9 shows this breach and the reaction between the cartridge and GaAs on the other visible areas on the wall.

## **CONCLUSION**

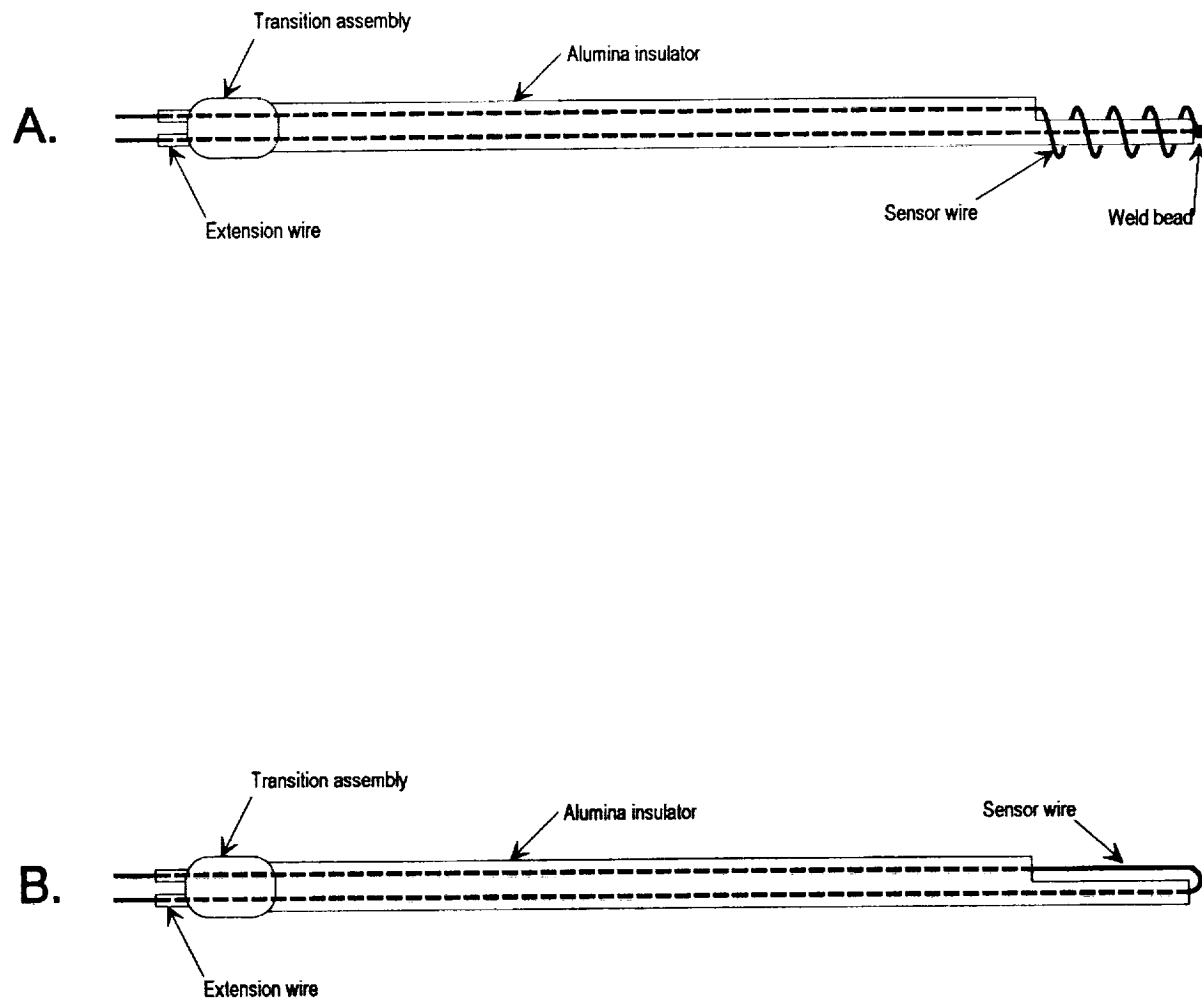
The two configurations of the failure sensors worked as designed. The helical wrapped failure sensor revealed an ampoule failure within 3.5 minutes. The flight configuration sensor showed an ampoule failure in 5.5 minutes. The faster reaction time of the helical wrapped sensor is due to the larger free surface area available for the reaction. In both experiments the cartridges were breached within 185 minutes after ampoule rupture.

In circumstances where space limitations are not critical, the helical wrapped design for the failure sensors is optimum due to its faster response time. However, the response time of the modified sensor that will be used in a GaAs experiment on the Second United States Microgravity Mission (USML-2) is adequate.



## REFERENCES

- [1] D. Watring and M. Johnson, Disclosure of Invention Report, NASA-MSF-28852 (1992).
- [2] D. Watring and M. Johnson, Ampoule failure sensor development for semiconductor crystal growth experiments, Journal of Crystal Growth, in press (1994).
- [3] M. Johnson and D. Watring, Ampoule Failure Sensor Time Response Testing-Experiment 1, NASA TM-108449 (1994).



**Figure 1. Configurations of ampoule failure sensors:**  
 (A) Helical loop-weld configuration;  
 (B) Single loop conductor configuration.

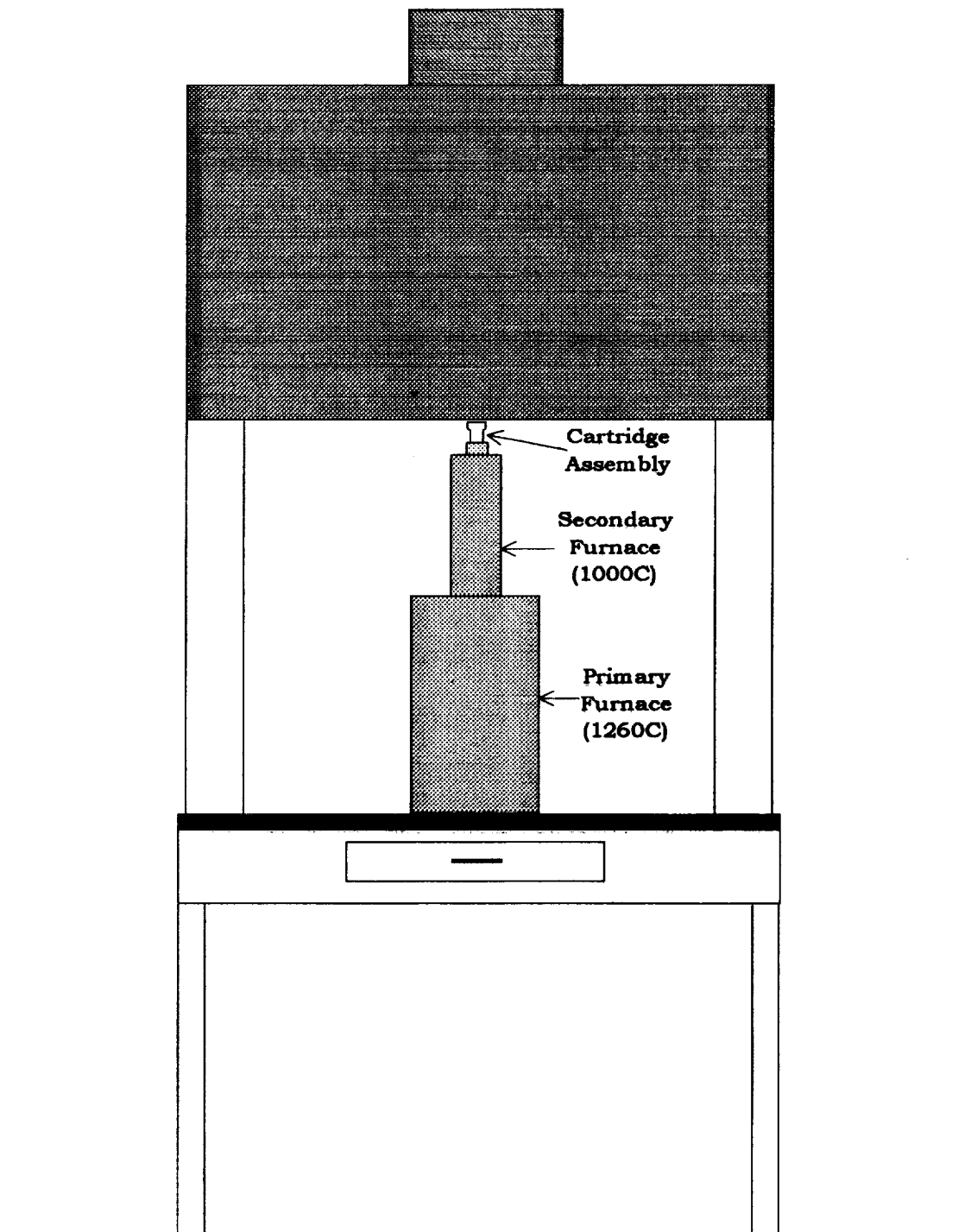


Figure 2. Furnace configuration.

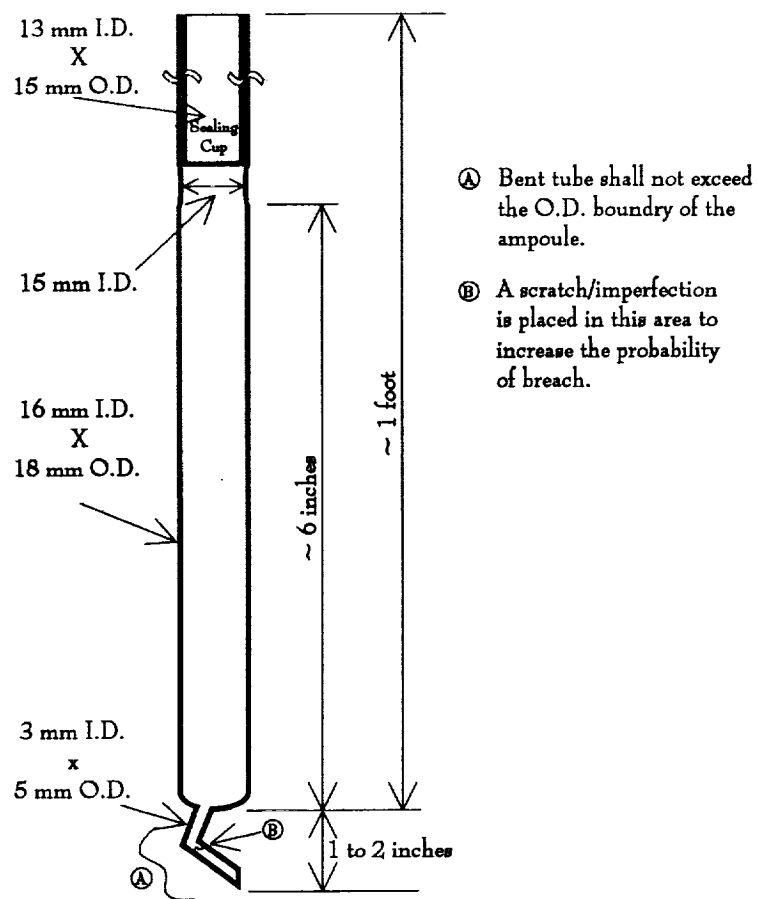


Figure 3. CGF breach test ampoule.

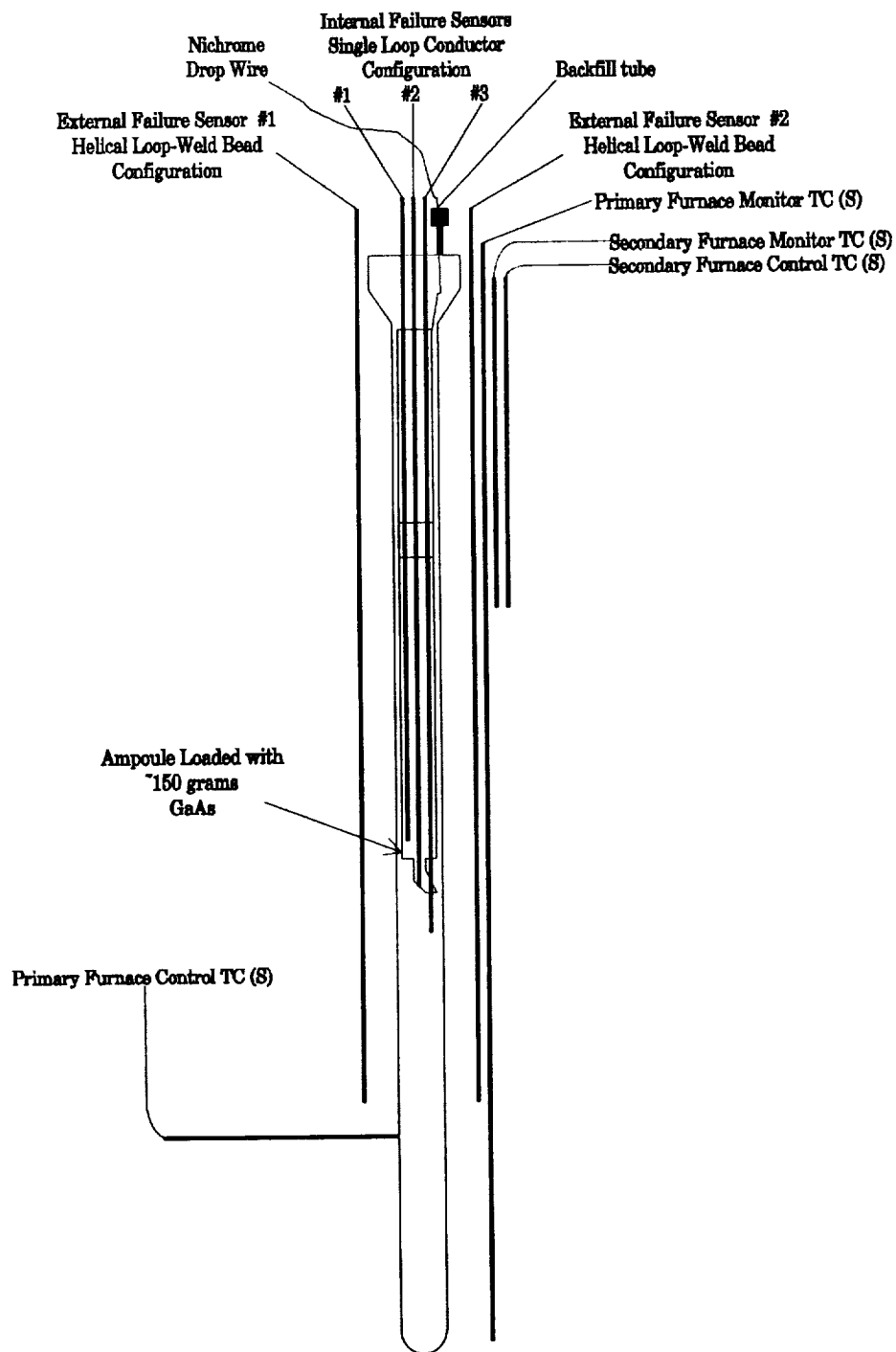


Figure 4. Pre-drop configuration, Experiment 3.

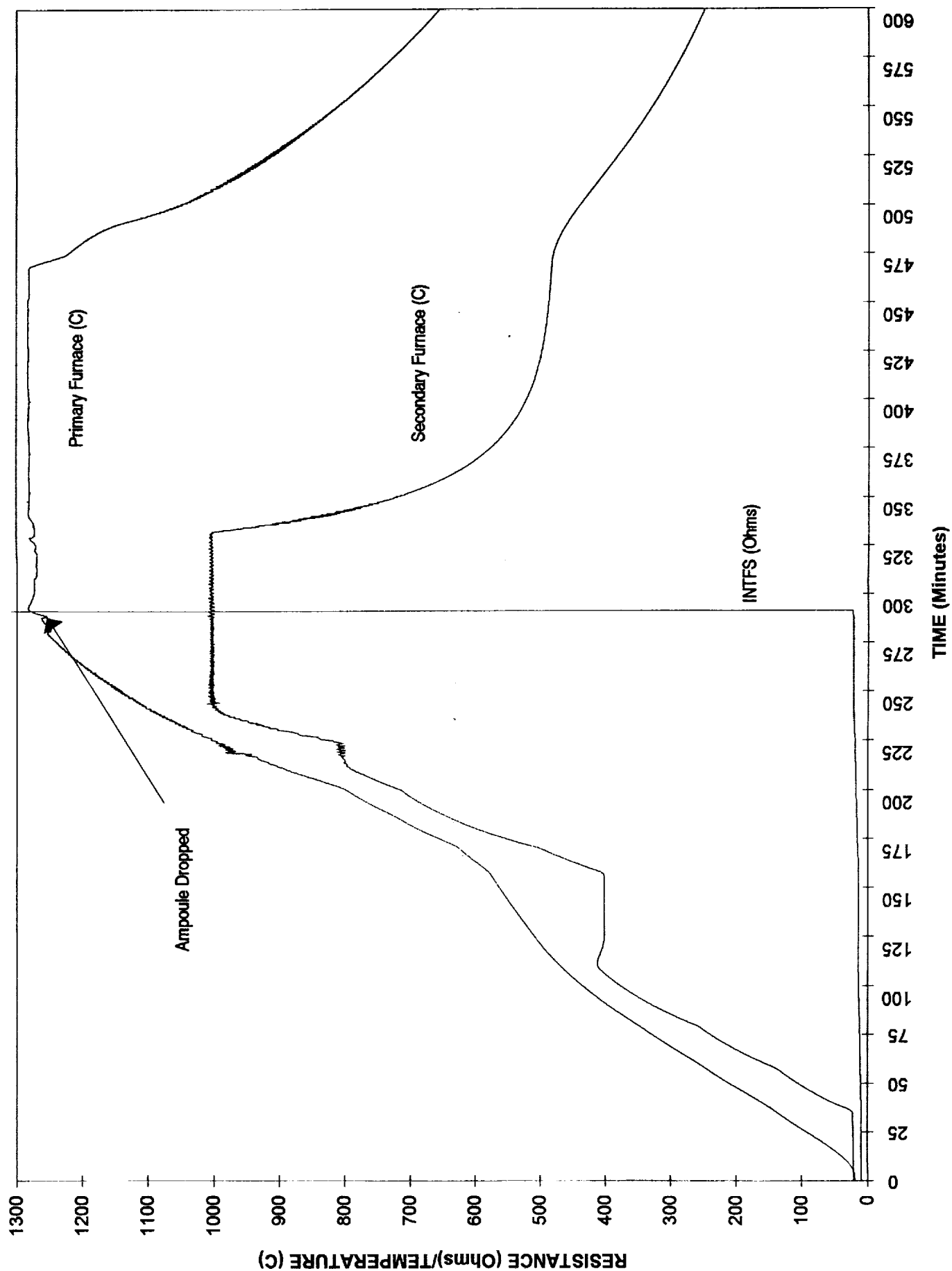


Figure 5. Ampoule failure sensor time response, Experiment No. 2.

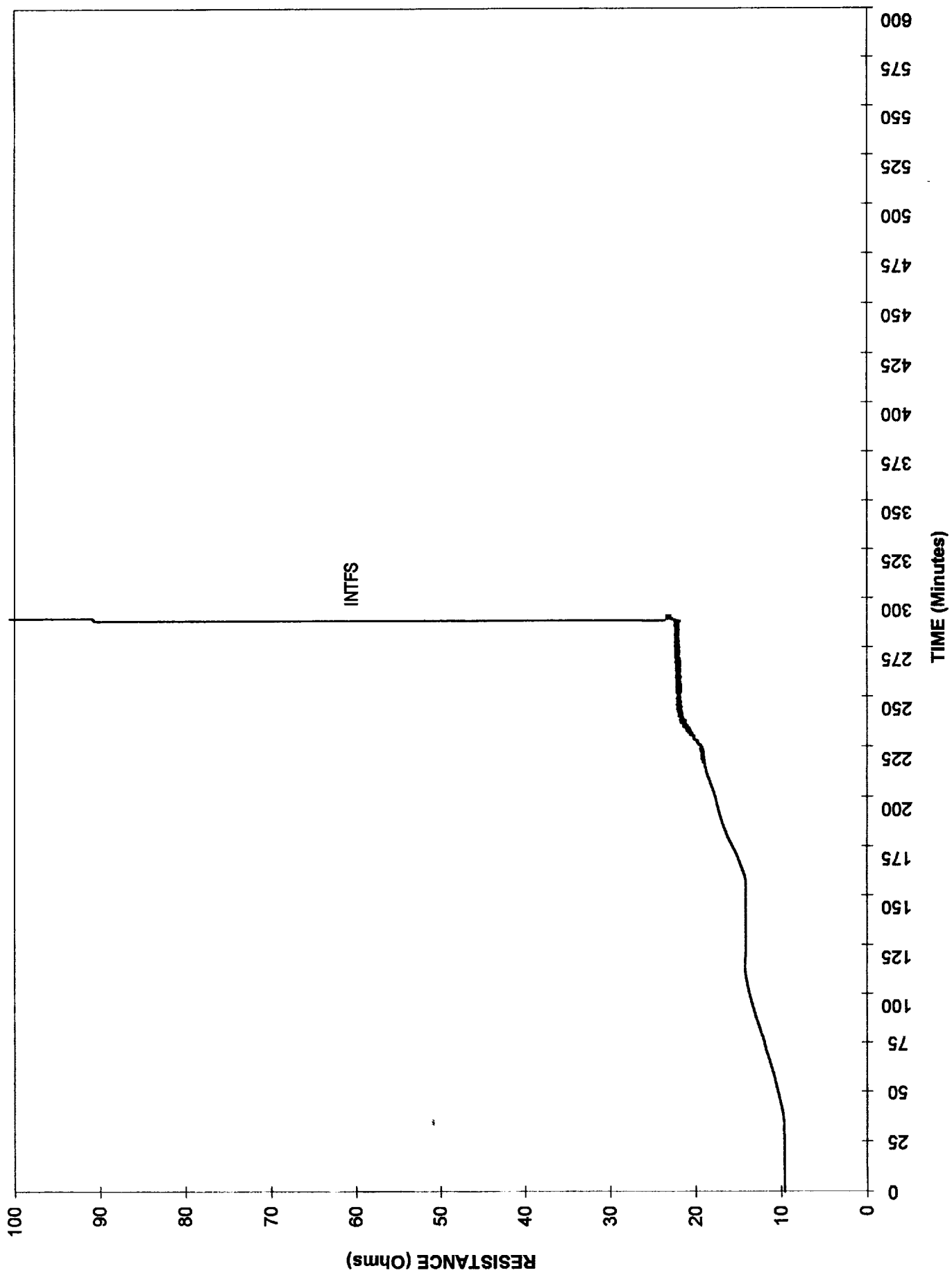
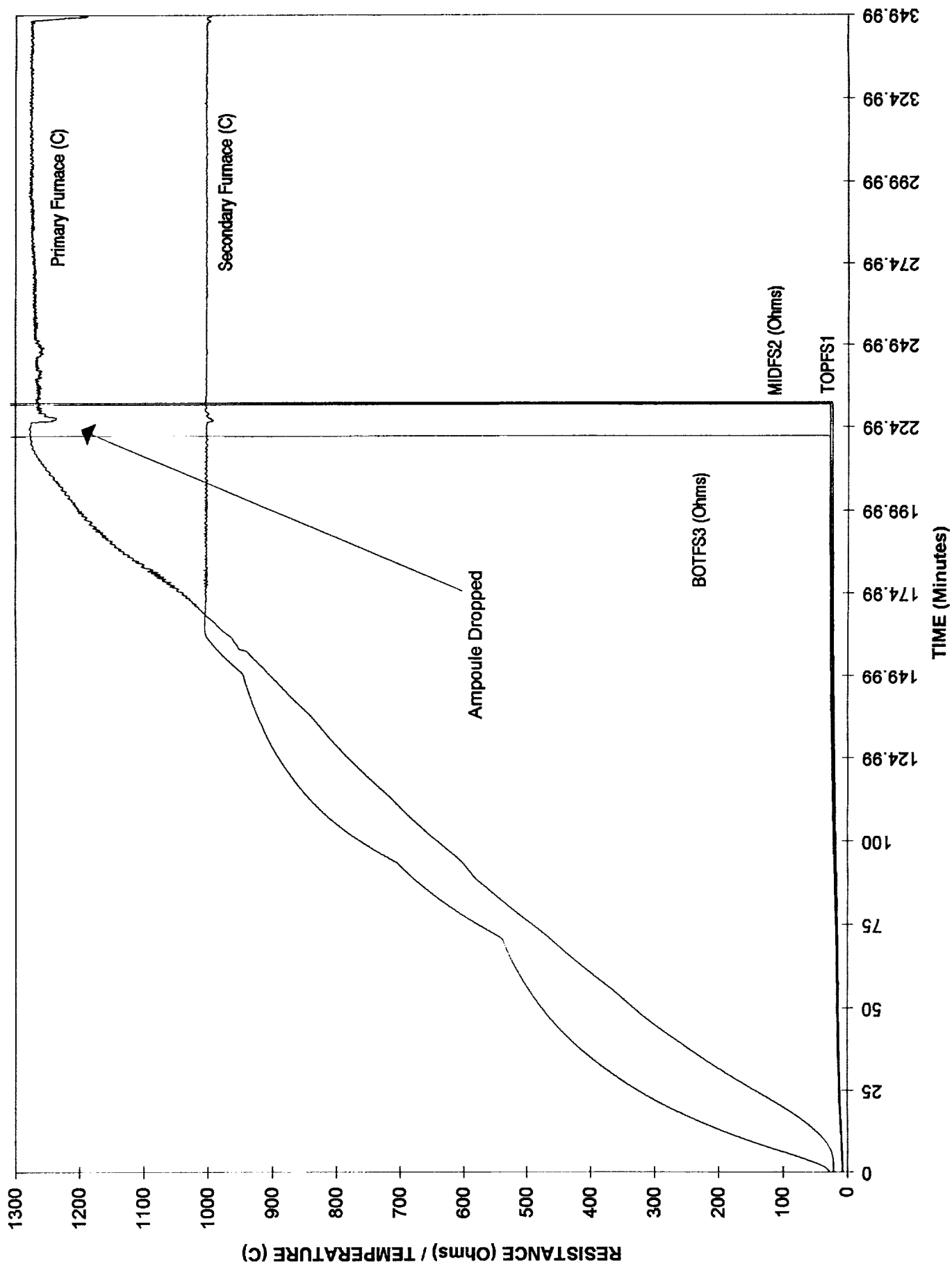


Figure 6. Detail of resistance vs. time for Experiment No. 2.





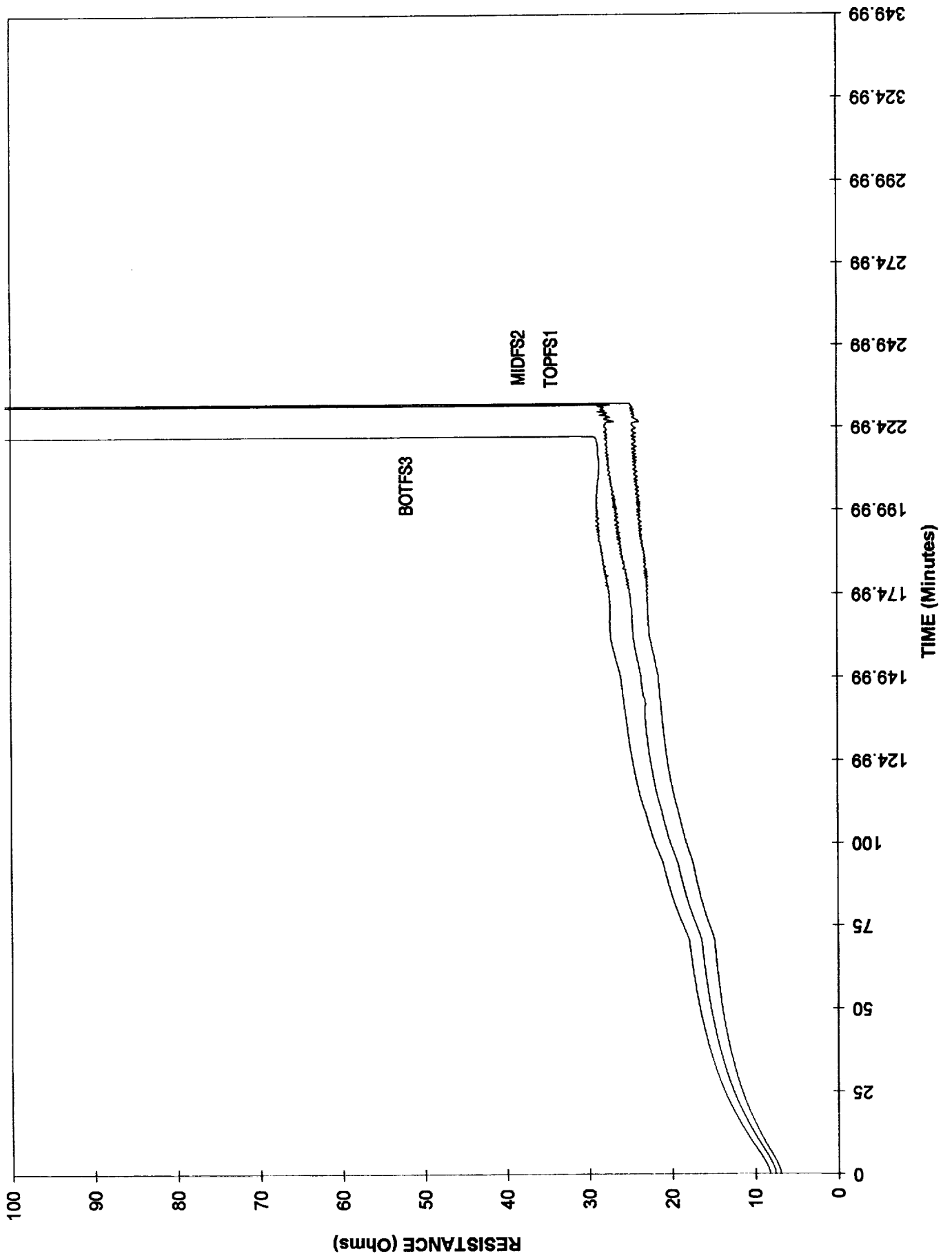


Figure 8. Detail of resistance vs. time for Experiment No. 3.



Figure 9. WC-103 cartridge breach, Experiment 3.

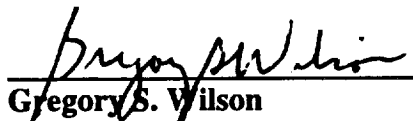
**APPROVAL**

**AMPOULE FAILURE SENSOR TIME RESPONSE TESTING—  
EXPERIMENTS 2 AND 3**

**By**

**M. L. Johnson and D. A. Watring**

**This report has been reviewed for technical accuracy and contains no information concerning national security or nuclear energy activities or programs. The report, in its entirety, is unclassified.**

  
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**Gregory S. Wilson**  
**Director, Space Sciences Laboratory**

**REPORT DOCUMENTATION PAGE**Form Approved  
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 1994		3. REPORT TYPE AND DATES COVERED Technical Memorandum	
4. TITLE AND SUBTITLE Ampoule Failure Sensor Time Response Testing— Experiments 2 and 3				5. FUNDING NUMBERS	
6. AUTHOR(S) M. L. Johnson and D. A. Watring					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) George C. Marshall Space Flight Center Marshall Space Flight Center, AL 35812				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, D.C. 20546				10. SPONSORING / MONITORING AGENCY REPORT NUMBER  NASA TM-108458	
11. SUPPLEMENTARY NOTES  Prepared by the Astrionics and Space Sciences Laboratory, Science & Engineering Directorate.					
12a. DISTRIBUTION / AVAILABILITY STATEMENT  Unclassified--Unlimited				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  The response time of an ampoule failure sensor exposed to a liquid or vapor gallium-arsenide (GaAs) and the corresponding breach time of the containing cartridge is investigated. The experiments were conducted in niobium-hafnium (WC-103) cartridges with an exterior silicide coating. These cartridges were built to flight specifications that were used in NASA's Crystal Growth Furnace during the first United States Microgravity Laboratory (USML-1) mission. The ampoule failure sensor is a chemical fuse made from a metal with which the semiconductor material reacts more rapidly than it does with the containing cartridge. In these experiments a platinum metal was used for the manufacture of the sensors. This technical report discusses the response time of two different sensor designs. The first design utilizes a helical wrapped wire and the second uses a single bare wire element. Experimental results indicate that both sensors are adequate in sensing the presence of molten or vapor GaAs with the latter having a 2-minute longer response time. In both experiments, the containing cartridge was breached within 185 minutes after ampoule rupture.					
14. SUBJECT TERMS  Ampoule Failure Sensor, Response Time, Gallium-Arsenide, Microgravity				15. NUMBER OF PAGES 20	
				16. PRICE CODE NTIS	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT Unlimited		